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Article

Modification of a Substrate Roughness for a Fabrication of Freestanding Electroplated Metallic Microstructures

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Abstract. This study aims to demonstrate a simple fabrication technique of freestanding electroplated metallic microstructures by modifying a substrate roughness. The proposed technique utilizes counter effects between two forces, i.e. an intrinsic force causing shrinkage in an electroplated metallic microstructure, and an adhesive force adhering a metallic microstructure to a substrate. With the modification of substrate roughness until the adhesive force becomes weaker than the induced intrinsic force, electroplated metallic microstructures would spontaneously release from the substrate after the electroplating process. Three parameters, i.e. substrate roughness, electroplated square structure's area and electroplated rectangular structure's width-to-length ratio, were experimentally studied. The results showed that the electroplated structure with a smaller size and smaller width-to-length ratio was more easily detached from the substrate for a given substrate roughness. In addition, for the same electroplated structure, a substrate with less roughness allowed a detachment of electroplated microstructure more easily.

Keywords: Freestanding, metallic structure, surface roughness, electroplating, copper, surface modification, photoresist mold.

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1. Introduction

For few decades, a nano and micro fabrication technology has played an important role in a manufacturing of Micro Electromechanical Systems (MEMS) as well as a fabricating in an emerging nanoscale science and engineering [1]. These applications are expected to significantly improve a quality of human life in many ways from electronic gadgets, healthcare and medical devices to future energy sources. Some examples of commercial MEMS products include MEMS accelerometers employed in automobiles and consumer electronic devices [2-3], digital micro-mirror devices for display applications in projectors and televisions [4-5], and MEMS pressure sensors for detecting pressures in car tires and blood vessels [6-7]. Functional elements of these devices often require thick metallic microstructures such as actuating and sensing units driven by electrostatic and electromagnetic principles. Among many manufacturing techniques, an electroplating is commonly used to deposit the thick metallic microstructures on a substrate due to its capability to fulfill high aspect ratio requirement, simplicity of fabrication and employability of inexpensive tools and chemicals. Hence, the electroplating technique is often chosen for low-cost MEMS mass production [8-9].

For various applications, such as membrane actuator [10], micro gear [11]), micro-scaled stenciling mask [12] or human body embedded sensor [13], not only a metallic but also freestanding microstructure is essentially needed. The metallic structure must be completely released from the substrate after electroplating process. Various techniques are currently used, for example, a sacrificial layer removal using a chemical etching or laser release [14-16], an abrasive removal using a grinding or polishing [17-18], and a removable printing mold [19]. However, these techniques are sometimes complex, destructive and expensive. Moreover, they might also introduce some limitation. For example, for freestanding metallic microstructures fabricated using a sacrificial layer and released from the microstructures by chemical etching, only a few metals are compatible with this sacrificial removal process resulting in a limitation of a type of metals for the metallic microstructures. Regarding this issue, a low-cost, simplicity and high-throughput technique for fabricating and releasing the metallic electroplated microstructure from the substrate is thus truly needed.

In this paper, we presented a simple methodology to fabricate freestanding metallic microstructures without hazardous process, specially required chemical or equipment to release completed structures. It is well known technique which starts with a stainless steel plate. The plate is then mechanically polished, chemically cleaned and is passivated in sodium chromate–nitric acid solution to facilitate easy removal of deposit from the plate [20-22].

The key difference of our proposed methodology is to modify substrate-surface's roughness without any chemical treatment before performing the electroplating process. This idea is inspired from the past work of Basrour & Robert [23]. In their work, an X-ray characterization of residual stress in an electroplated nickel showed that the electroplated metallic structure would have in-plane intrinsic stresses developed during the electroplating process, and, for a rectangular structure, the intrinsic stress in a lateral direction would be larger than that in a longitudinal direction.

In principle, the proposed methodology utilized counter effects between two forces, i.e. an intrinsic force inducing shrinkage in metallic microstructures, and an adhesive force adhering metallic microstructures to the substrate. A magnitude of the intrinsic force depends on both amplitude of aforementioned intrinsic stress developed during the electroplating and a side-edge surface area, i.e. a thicker electroplated structure with rectangle's longer side length has the larger total intrinsic force for the same intrinsic stress. On the other hand, a magnitude of the adhesive force depends on both amplitude of adhesive stress and a contact-surface area between the substrate and electroplated material, i.e. a larger contact-surface area has a larger total adhesive force for the same adhesive stress. However, for a given square or rectangular structure, the effective contact-surface area would be increased with the increment of surface roughness, and vice versa. Therefore, with the modification of the roughness of substrate for electroplating until the magnitude of adhesive force is weaker than that of the intrinsic force, metallic microstructures would be spontaneously detached from the substrate after the electroplating process.

This work has experimentally investigated the effects of a substrate roughness and a shape of electroplated structures on a detachment of freestanding copper microstructures after the electroplating process. This fabrication methodology would be promising for various applications since copper exhibits properties of low resistance, high current carrying capability and scalability which promote its ability to support high current density and viability for micro-devices [24-28].

2. Sample Preparation and Experiment Conditions

The substrate employed in this study was a 10x10 cm² stainless-steel plate. It was cleaned and polished with abrasive papers of different grit sizes using a machine called METASERV 2000 Grinder/Polisher at the speed of 150 rpm for 1 hour and 30 minutes for all experiments. A roughness of stainless-steel substrate after polishing was then measured by Surftest Mitutoyo SV3000. The sensor tip was drawn on the substrate for a straight ten-millimeter line at four different positions perpendicular to a polished trace. The averaged roughness of the stainless-steel substrate after polishing was equal to 57, 29, 22 and 12 nm with an uncertainty between each polishing for the same experiment conditions within ± 5 nm.

After polishing, the substrate was rinsed with clean water and dried. The negative photoresist film with thickness of 40 μm was then laminated on the polished substrate using a heating roller at 90°C for three times. After that, the designed mask was placed on the photoresist film, and the substrate together with the mask was then exposed to UV light source for 20 seconds. After the UV light exposure, the substrate was developed in sodium carbonate solution, 3 g Na₂CO₃/300 ml H₂O, with consistently shaking to facilitate the removal of unexposed part. This was followed by rinsing with clean water and baking at 120°C for 7 minutes with a hotplate to help evaporating leftover solvents. After this process, the fabrication of photoresist mold on the stainless substrate was finished.

Subsequently, the stainless-steel substrate with patterned photoresist was dipped in copper sulfate, CuSO₄, as electrolyte solution to deposit a copper film inside the fabricated mold by an electroplating process. After finishing depositing to a desired copper thickness, the negative photoresist on the substrate was removed with sodium hydroxide solution, and electroplated copper structures were naturally detached as expected. Figures 1a-c depict a fabrication process flow: polishing, lithography and electroplating as explained earlier.

Two sets of experiment with different electroplated structure shapes, i.e. square and rectangular structures, were conducted in this study. For the first experiment, the square structures with different sizes of 3.2x3.2, 4.5x4.5, 6.3x6.3, 7.8x7.8, 8.9x8.9 and 10x10 mm² were tested while the average roughness of substrate were varied as 57, 29, 22 and 12 nm. For the second experiment, the size of all rectangular structures was approximately kept at 40 mm² while the width and length were varied as 1x40, 2x20, 4x10, 5x8 and 6.3x6.3 mm². With these dimensions, the ratios of width-to-length corresponded to 0.025, 0.1, 0.4, 0.63 and 1.0, respectively. However, they were tested only with the average roughness of substrate equal to 29 and 22 nm. For all cases, three tests, with ten samples for each test, were conducted. Figures 2a and b respectively illustrate shapes of tested copper structure for both sets of experiment, and an image taken while electroplated copper structures detaching from the substrate in the experiment.

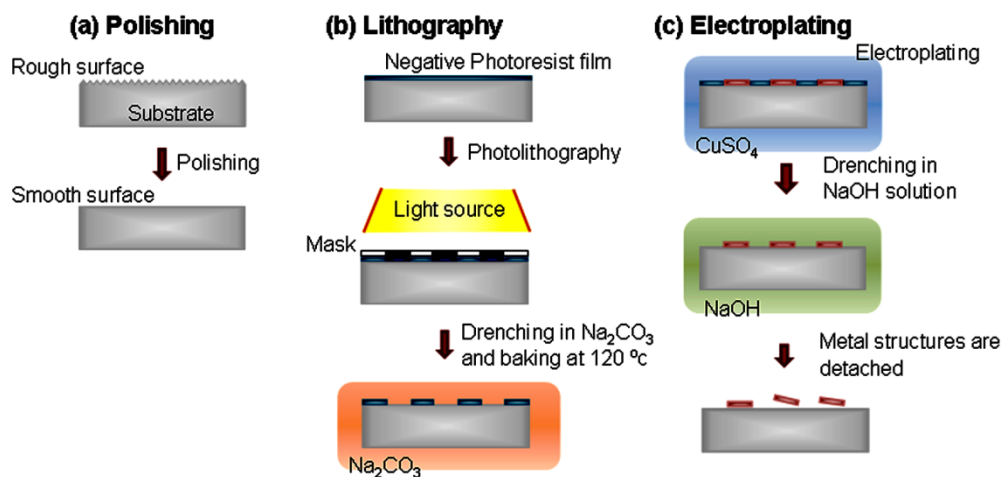


Fig. 1. Process flow of the fabrication process: (a) polishing a stainless-steel substrate, (b) constructing a photoresist mold, (c) electroplating metal inside the mold.

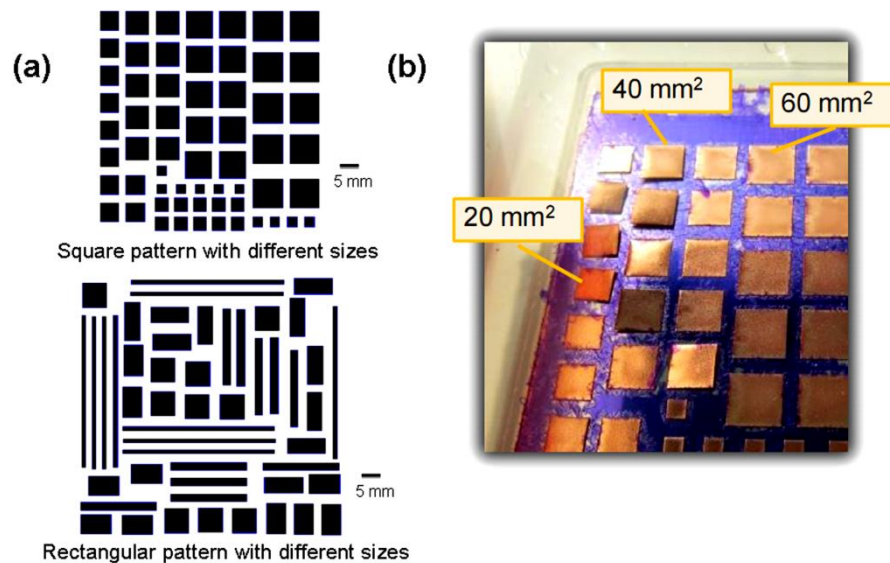


Fig. 2. Electroplated copper structures: (a) square and rectangular patterns, (b) taken image of detached copper structures during the releasing process.

Moreover, the electroplated copper thickness was fixed in order to keep the intrinsic stress, due to the effects of thickness, constant for all experiments. Despite of our attempts, the copper thicknesses were slightly different due to a difficulty to control uniformity of electric field while electroplating as a result of a slight variation of structure thickness for the same experiment conditions. The thickness of copper microstructures was approximately equal to 138.8 ± 5 and 140.5 ± 5 μm for the first and second experiment, respectively. It should be noted that applied current for both experiments was kept constant at 0.9 A that resulted in slightly dissimilar current density due to the difference in an electroplated surface area between two experiments. The current density was approximately equal to 29 and 32 mA/cm^2 for the first and second experiment, respectively.

3. Results and Discussions

After the end of fabrication for each experimental case, the number of detached structure was counted comparing to the total number of metallic freestanding structure (thirty samples). A detachment ratio, the ratio between the number of detached structures and that of all structures for the same dimensions, was then calculated for each condition. The detachment ratio equal to zero means no detachment for all microstructures of the same experiment conditions is found. On the other hand, if the microstructures of the same experiment conditions are totally detached, the detachment ratio is equal to one. It should be noted that an incomplete detachment during the fabrication could occur due to the non-uniformity of surface roughness, current density as well as electroplated structure's thickness.

3.1. Square Shape with Different Structure Sizes

Figure 3 shows an average detachment ratio of microstructures for different substrate roughness and area of the square electroplated-structure. From the experiment results, it was found that the variation of detachment ratio among tests was quite large for some experiment conditions resulting from a non-uniformity of the substrate's roughness and the electroplated structure's thickness as mentioned earlier. For the roughness of 57 nm that was the roughest condition, a detachment of copper microstructure was rarely found after removing the photoresist for all structure sizes. This was attributed to considerably large effective contact surface area due to large substrate roughness as a result of a significantly large adhesive force compared to an intrinsic force. However, for smoother substrate surface, a relatively higher detachment ratio was observed. For example, the detachment ratio increased up to 0.5 for the substrate roughness of 29 nm and the square structure's sizes of 3.2×3.2 mm^2 , or the structure's area of approximately 10 mm^2 . On the other hand, for a fixed substrate roughness, the detachment ratio gradually

decreased for larger square structure's sizes. In addition, when the substrate roughness reduced to 12 nm, the electroplated structures for all structure sizes were almost totally detached from the substrate. This was likely attributed to a considerably small effective contact surface area that resulted in a relatively small adhesive force compared to an intrinsic force for all structure sizes. Hence, the experiment results showed that a smoother substrate surface and a smaller square microstructure had a tendency to enhance a possibility of detachment of electroplated metallic structures.

From the experiment results, it was shown that both roughness of stainless-steel substrate and size of electroplated microstructures significantly affected the detachment of electroplated microstructures. With combining these two parameters, a new parameter that was a multiplicity between the substrate roughness and the electroplated structure's area was introduced. This new parameter represented the effective contact-surface area between the substrate and electroplated structure, and the adhesive force should reduce if this new parameter became lower.

Figure 4 shows the relationship between this multiplicity parameter and the detachment ratio. The result suggested that the electroplated copper microstructure with the manufacturing conditions as in this experiment would tend not to detach freely if the substrate's roughness multiplied by the electroplated copper microstructure's area was higher than approximately $5 \times 10^{-4} \text{ mm} \times \text{mm}^2$. In addition, when this multiplicity parameter increased, the possibility of detachment of electroplated structure became lower as expected.

Furthermore, the increase of electroplated microstructure's size would affect both intrinsic and adhesive stresses as follows. If a square structure's size was increased by two times for a given thickness and substrate roughness, the side-edge area of the electroplated structure (surface B and C in Fig. 5a) as well as the intrinsic force should be increased by two times, while the effective contact surface area (proportion to surface A in Fig. 5a) as well as the adhesive force should be increased by four times, and so on, as depicted in Fig. 5a. Thus, it was more difficult for a larger square structure to peel off due to a drastically increase of the adhesive force comparing to the intrinsic force for the same substrate roughness. This idea was in good agreement with the experiment results as explained earlier. On the other hand, the roughness modification affected only the adhesive force due to a change of effective contact-surface area. Thus, a smoother substrate that corresponded to a smaller effective contact-surface area as well as a lower adhesive force had a tendency to enhance a possibility of detachment of electroplated metallic structure for a given electroplated structure size.

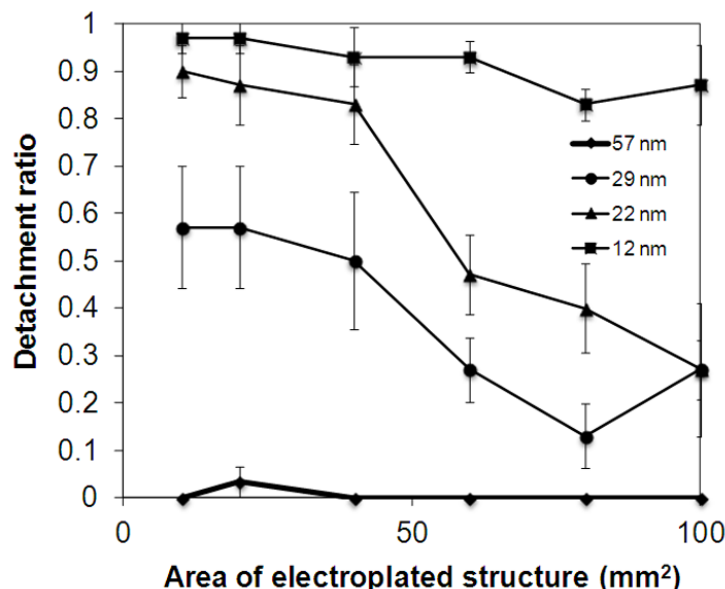


Fig. 3. Detachment ratio for different sizes of square-shaped metallic structures and substrate roughness.

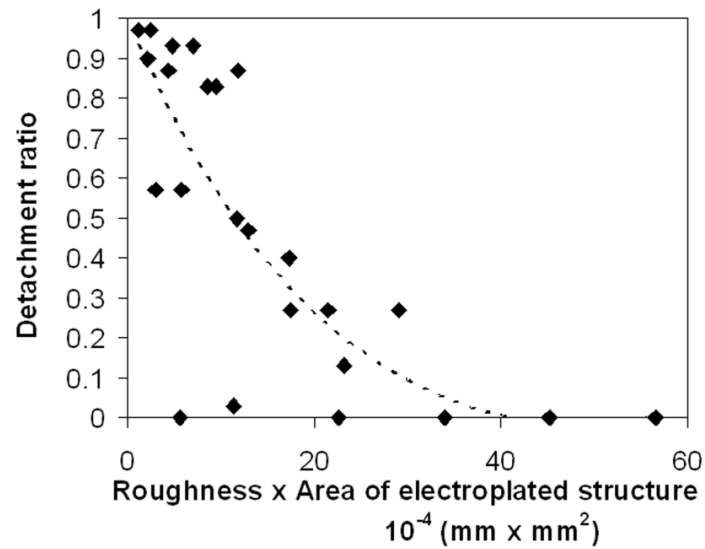


Fig. 4. Effect of the multiplicity parameter between the substrate roughness and the structure surface-area on the detachment ratio.

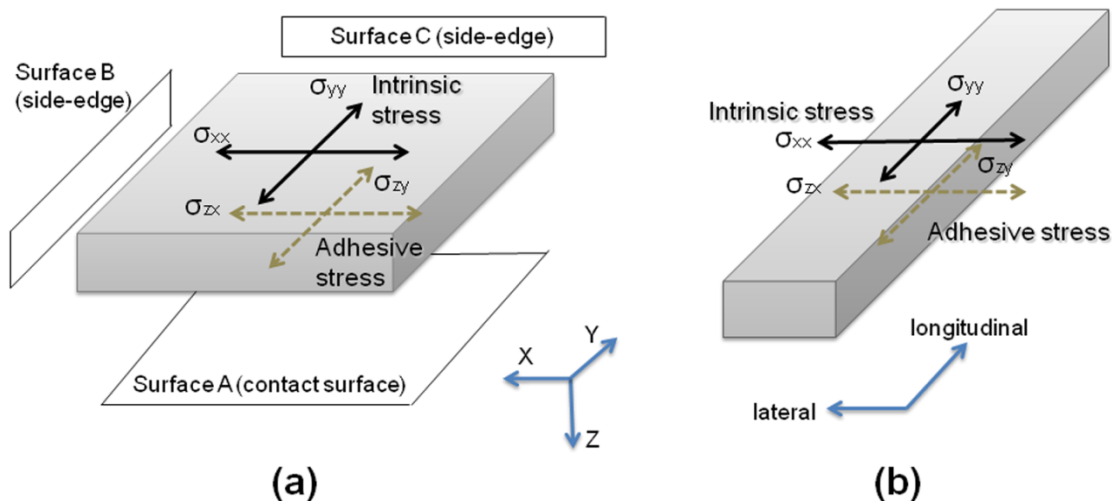


Fig. 5. Intrinsic and adhesive stresses in the electroplated microstructures: (a) square structure ($\sigma_{xx}=\sigma_{yy}$), (b) rectangular structure ($\sigma_{xx}>\sigma_{yy}$).

3.2. Rectangular Shape with Different Width-to-Length Ratios

Figure 6 shows an average detachment ratio of microstructures for different substrate roughness for various rectangular structure's width-to-length ratios. For the substrate roughness equal to 22 nm, all width-to-length ratios of rectangular structures, except the width-to-length ratio equal to one, or square structure, were completely detached. On the other hand, for a rougher substrate with its roughness equal to 29 nm, the detachment ratio was varied for different width-to-length ratios. With the width-to-length ratio of 0.025 (width : length = 1:40), or utmost slim structure, the detachment ratio reached one or all structures were completely detached. In addition, the detachment ratio gradually decreased when the width-to-length ratio became higher.

In this experiment, all cases had the same structure's size of 40 mm², hence, with a given substrate roughness, the effective contact surface area as well as adhesive force was constant. However, when varying the width-to-length ratio, the possibility of detachment changed that should be the results of the variation

of width-to-length ratio. From Basrour & Robert [23], the difference in lengths of two perpendicular sides, i.e. width and length, of the rectangular structure resulted in different induced intrinsic stress. The intrinsic stress acted perpendicular to the longitudinal side (σ_{xx}) tended to be 5-10% larger than that perpendicular to the lateral side (σ_{yy}). This locally higher intrinsic stress perpendicular to the longitudinal side, conceivably together with a larger lateral side-edge area, resulting in a higher magnitude of intrinsic force in the lateral direction of rectangular structure as depicted in Fig. 5b, would make it easier to have the electroplated structure peel off. In addition, the variation of width-to-length ratio should introduce the change of stresses in both longitudinal and lateral directions as well. From the experiment results, a metallic structure with a smaller width-to-length ratio was more easily detached from the substrate when the substrate's roughness and structure's size were fixed. The results implied the larger intrinsic stress was induced on a structure with the smaller width-to-length ratio.

4. Applications

As shown in our experiment results, this fabrication methodology was very simple and promising for many applications. To demonstrate its utilization, the proposed fabrication technique was employed to construct different shapes of freestanding metallic elements for two actuators of different actuating principles, i.e. electromagnetic and shape-memory alloy actuators. Figure 7a illustrates a flexible membrane embedded with a copper coil of an electromagnetic actuator. The micro coil was made of copper with a width of 500 μm and a thickness of 50 μm by this proposed technique, and later on assembled to a polymer membrane with a thickness of 250 μm . When assembled as the membrane with a pre-straining of 10%, the actuator whose diameter was equal to 20 mm exhibited a large deflection of 25 μm when DC current of 3.2 A was applied. In addition, the membrane actuator showed a resonant frequency approximately at 5 Hz.

Figure 7b illustrates a cantilever shape-memory alloy actuator whose base structure was previously made of copper, and, nickel titanium (NiTi) was then deposited on the top of the copper structure. The copper structure had a width of 250 μm and a thickness of 100 μm , while the deposited NiTi had a thickness of 5 μm . The 20-mm long cantilever structure showed a large deflection up to 300 μm when DC current of 5 A was applied; however, its response was quite slow in an order of 10 seconds. Detail of this work is in Ref. 29.

In addition to these two applications, the proposed methodology would be suitable for various kinds of work. Example is a sensing element for an electrochemical sensor, a shadow mask for a thin-film deposition and a micro-capillary structure for a filtration. Other demonstrations of this fabrication technique to construct a real micro-device are in Refs. 30-31.

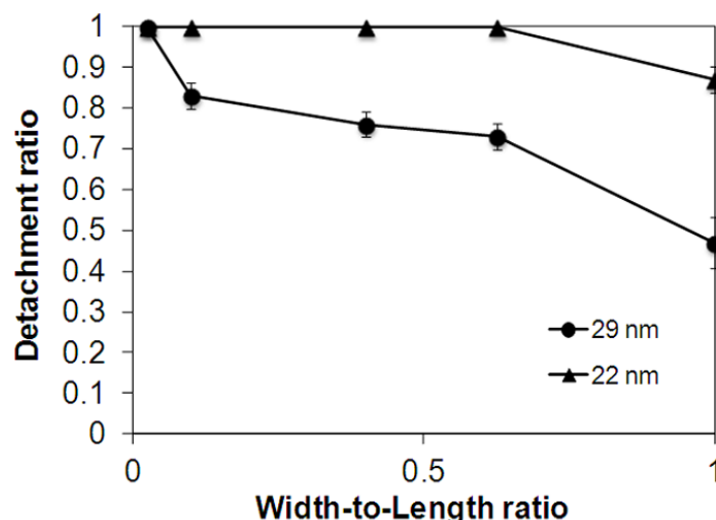


Fig. 6. Detachment ratio for rectangular-shaped metallic structures with different width-to-length ratios and substrate roughness.

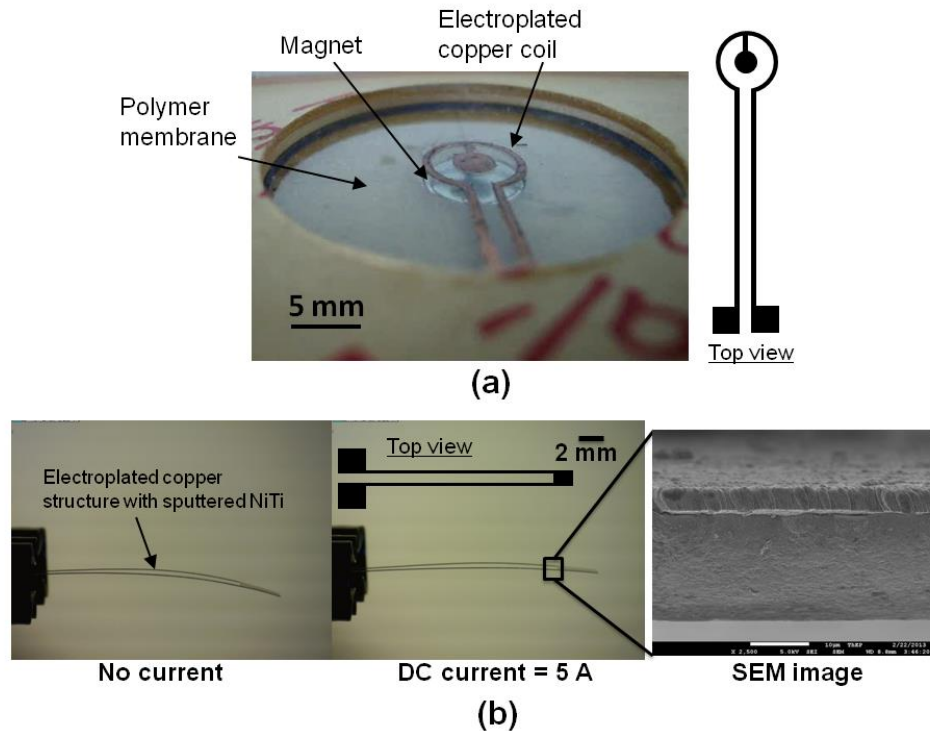


Fig. 7. Examples of freestanding electroplated metallic elements for a micro-actuator application: (a) micro coil of an electromagnetic membrane actuator, (b) bimorph structure of a shape-memory alloy actuator.

5. Conclusions

This study aims to demonstrate a simple fabrication technique of freestanding electroplated copper microstructure by modifying the substrate roughness. With the modification of substrate roughness until a magnitude of adhesive force locking a metallic microstructure to the substrate becomes weaker than that of induced intrinsic force inside the microstructure during the electroplating, deposited metallic microstructures will be spontaneously released from the substrate after the electroplating process. The fabrication technique started with a polishing of a stainless-steel substrate following by a patterning of a photoresist mold of microstructures. The copper was then electroplated inside the mold until a desired thickness was achieved. After that, the copper microstructure was released when removing the photoresist mold. Two experiments were conducted to investigate the effects of substrate roughness simultaneously with electroplated structure's size and electroplated structure's width-to-length ratio on a detachment possibility. From both experiments, the results showed that the electroplated structure with a smaller size and smaller width-to-length ratio was more easily detached from the substrate for a given substrate roughness. In addition, for the same electroplated structure, a substrate with less roughness allowed a detachment of electroplated structure more easily. However, both substrate's roughness and electroplated structure's size should be small enough in order to have the electroplated structures peel off. From the experiment results, if the multiplicity between the substrate's roughness and the electroplated structure's area was smaller than $5 \times 10^{-4} \text{ mm} \times \text{mm}^2$, the detachment possibility of electroplated structure from the substrate became very high.

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